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Sampling and Analysis Protocol for Assessing Organic Emissions from Stationary Combustion Sources in Exposure Evaluation Division Combustion Studies

Midwest Research Inst., Kansas City, MO

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METHODS MANUAL

Sampling and Analysis Procedures for Assessing Organic Emissions from Stationery Combustion Sources in Exposure Evaluation Division Studies

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16. ABSTRACT

The sampling and analysis methods described in this report were specifically designed for use in an ongoing nationwide survey of emissions of organic pollutants from stationary combustion sources. The primary focus of this survey is on polynuclear aromatic hydrocarbons (PAHs) and polychlorinated aromatic hydrocarbons including polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins (PCDDs), and polychlorinated dibenzofurans (PCDFs). To date, these procedures have been used by Midwest Research Institute (MRI) to survey emissions from coal-fired utility boilers, a co-fired (coal + refuse-derived fuel) utility boiler, and a municipal refuse incinerator. This document was prepared by MRI as a guideline for laboratories who may participate in this study, and for other researchers who wish to use these methods.

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SAMPLING AND ANALYSIS PROTOCOL FOR ASSESSING ORGANIC EMISSIONS FROM STATIONARY COMBUSTION SOURCES IN EXPOSURE EVALUATION DIVISION COMBUSTION STUDIES

by

John S. Stanley
Clarence L. Haile
Ann M. Small
Edward P. Olson

Midwest Research Institute
425 Volker Boulevard
Kansas City, Missouri 64110

METHODS MANUAL

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Prepared for:

U.S. Environmental Protection Agency
Office of Pesticides and Toxic Substances
Field Studies Branch
401 M Street, S.W.
Washington, D.C. 20460

Attn: Dr. Frederick Kutz, Project Officer
Mr. David Redford, Task Manager

PREFACE

This sampling and analysis document was prepared for the Environmental Protection Agency under EPA Contract No. 68-01-5915. The methods described in this document were designed for use by Midwest Research Institute in assessments of stationary combustion source emissions. They may also be used as guidelines by other researchers who wish to conduct comparable studies. This document was prepared by Dr. John S. Stanley, Dr. Clarence L. Haile (MRI Task Manager), Ms. Ann M. Small, and Mr. Edward P. Olson.

MIDWEST RESEARCH INSTITUTE

John E. Going
Program Manager

Approved:

James L. Spigarelli, Director
Analytical Chemistry Department

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SECTION 1

INTRODUCTION

The sampling and analysis methods described in this report were specifically designed for use in an ongoing nationwide survey of emissions of organic pollutants from stationary combustion sources. The primary focus of this survey is on polynuclear aromatic hydrocarbons (PAHs) and polychlorinated aromatic hydrocarbons including polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins (PCDDs), and polychlorinated dibenzofurans (PCDFs). To date, these procedures have been used by Midwest Research Institute (MRI) to survey emissions from coal-fired utility boilers, a co-fired (coal + refuse-derived fuel) utility boiler, and a municipal refuse incinerator. This document was prepared by MRI solely as a guideline for other laboratories who may participate in the same study, and for other researchers who wish to use these methods.

SECTION 2

SAMPLING

The procedures described in this section have been used by MRI in obtaining representative samples of the inputs and the emissions from stationary conventional combustion sources. These procedures encompass the specific requirements for site surveys, pretest preparations, and actual sampling procedures. A quality assurance program and sample control and custody documentation procedures are also presented.

The precise sampling procedures used for a specific plant may vary somewhat depending on the specific configuration and operation of the facility. The samples that should be collected include gaseous, solid, and liquid materials. Gaseous emissions should be collected by the EPA Method 5 procedures[1] modified for the capture of trace organic compounds as described in this section. The solid and liquid samples should be collected according to a sound, statistically designed 24-hr schedule.

These methods were designed to provide both qualitative and quantitative information on polycyclic and chlorinated organic compounds. Therefore, it is imperative that the sampling procedures should be followed as closely as possible to prevent contamination or compromise the integrity of the samples.

MEDIA SELECTION

The sample collection program should be designed to allow accurate assessments of the organic pollutants in both input to and emissions from the combustion process. Although the focus of this project is on organic compounds that likely undergo considerable chemical changes in the combustion process, the media selection criteria are the same as would be used to determine a mass balance for a conservative pollutant, e.g., a nonvolatile metal. It is of paramount importance that the collection procedures provide the most representative specimens of the media selected.

The specific media and sampling points for each plant will depend somewhat on the specific design of the plant. However, the media can be described in three categories: inputs, emissions, and miscellaneous media. The principal inputs to the combustion process are fuels and combustion air. All primary fuels should be sampled. Fuels used only for unit startup should be excluded from the sampling program. Combustion air can be sampled near the air intake for the unit. However, care should be taken to avoid collection of fugitive dust that may be suspended by the activities of the sampling equipment or personnel.

The principal emission media are the bottom ash, i.e., the residue from the combustion process, and the flue gases with associated fly ash. Flue gas emissions may have the most widespread impact on the quality of the surrounding environment. Hence, flue gas samples must be collected at a point downstream of the unit's particulate emissions control systems. However, the materials collected by air pollution control devices must also be sampled to allow accurate characterization of the total plant emissions. For example, many coal-fired utility boilers employ electrostatic precipitators (ESPs) to control particulate emissions. The fly ash collected in the ESP hoppers of these plants must be included in the sampling program. In addition, ash that is removed from flue gases by economizers in many plants must also be sampled where practical.

Miscellaneous sample media include other materals that may have direct contact with the combustion products. Examples are the input and overflow waters from a bottom ash wet quenching system. These secondary emission media may pose environmental hazards depending on the plant disposal practices.

A list of typical sample capture requirements for several materials is given in Table 1. Included are sample size, storage container type and size, sampling frequency, and total samples obtained each day. No compositing should be done in the field. All samples should be placed in the appropriate containers prepared as outlined in the pretest preparation and setup procedures.

PRESAMPLING SITE VISIT

Representatives from EPA and the sampling crew chief must consult with the plant supervisor to determine where and how each type of sample may be captured. The crew chief should obtain data on key parameters related to flue gas sampling. These parameters include the stack dimensions, flue gas temperatures, moisture content, static pressure, and flue gas velocities.

Sampling points for grab samples should be located as close as possible to the actual combustion process to avoid sampling combined streams (e.g., from multiple units) or combined waste media (e.g., ash-water mixtures) and to prevent dilution of the desired sample. These precautions should allow simpler data assessments. It is also advantageous to centralize the sampling locations if possible such that the sampling schedules can be followed accurately by the sampling crew. Where possible, special aids for obtaining the samples in a safe and efficient manner should also be considered. Plant staff-operated equipment, limited access areas, special tools, electrical outlets, and periodic safety calls are some possible considerations.

Once all possible sampling points are determined, a statistically sound, random sampling scheme should be provided for solid/liquid sampling that corresponds with the flue gas sampling activities. The sampling schedule should be constructed to provide the sampling team with the specific time and location a sample will be taken. It may be necessary to follow an assigned grid pattern or port selection scheme in order to effectively subsample large surface areas. In addition, sampling schedules may be subject to change each day.

TABLE 1. SAMPLE CAPTURE REQUIREMENTS

Material	Storage size/type	Sample frequency	Total samples (24 hr)
Solid			
Coal	1 qt amber glass	Twice per shift	6
Refuse or RDF[a]	1 qt amber glass	Twice per shift	6
Bottom ash	1 pt amber glass	Twice per shift	6
Fly ash	1 pt amber glass	Twice per shift	6
Other solid waste	1 pt amber glass	Twice per shift	6
Gaseous			
Dry particulate	1 pt amber glass	One per train[c]	2
Reeve Angel 934 AH filter[b]	150 mm X 15mm glass petri dish	One per train[c]	2
Nozzel, probe, cyclone and flask combined rinses	1 qt amber glass (may require additional 250 ml of same)	One per train	2
Sorbent trap	Traps capped with plugs.	One per train	2
First impinger with rinses	950 ml amber glass (may require additional 250 ml of same)	One per train	2
Control 934 AH filter[b]	150 mm X 15 mm glass petri dish	One per day	1
Combustion air	Sorbent trap capped with plugs	One per day	1
Liquid			
Effluent water	1 qt amber glass	Twice per shift (duplicates)	12
Influent water	1 qt amber glass	One per day (duplicate)	1

a Refuse - derived fuel.

b Or equilvalent.

c Dry particulate will be collected only from trains using a cyclone trap. This trap may not be necessary if particulate loading is light.

d Additional filters may be necessary if particulate loading is high.

The presampling site visit should also allow the crew chief to determine local sources for expendable sampling supplies. In addition, the most convenient accommodations for the sampling crew during the testing period should be located.

FLUE GAS SAMPLING EQUIPMENT AND MATERIALS

The modified Method 5 train, shown schematically in Figure 1, is used to collect samples for organic compounds from the stack. (See Note below.) Additional empty impingers may be added just after the first impinger to retain water from high moisture gases. This train should be operated according to Method 5 specific procedures modified by the additional cleanup and recovery procedures required for organic compounds.

The sampling probe liners must be glass or TFE, depending on the flue gas temperature. A glass cyclone should be provided for high particulate gases to avoid excessive filter loading. Vaporous organics are collected by a sorbent trap (Figure 2). This trap is located in the sample line downstream of the heated oven and upstream of the first impinger. The trap is packed with precleaned XAD-2. The module that houses the sorbent trap is water-jacketed. Cold water from an ice bath surrounding the impingers is pumped through the jacket to maintain an outlet temperature of $\leq$ 16°C (60°F). Because of the possible sensitivity of potential analytes to ultraviolet light all sorbent traps should be kept wrapped in aluminum foil.

All solvents used for preparing the sampling train for testing and for field laboratory cleaning of sample trains should be stored in glass or TFE bottles. All solvents should be Burdick and Jackson Distilled-in-Glass or equivalent grade. TFE or stainless steel forceps should be used for handling filters. The train and train components that contact the sample should be handled with clean, bare hands, i.e., without gloves.

Flue Gas Sampling Pretest Preparation

All train components that will contact the sample (probe, cyclone, filter holder, resin cartridge, and connecting tubes) must be clean of all potentially interfering materials. Component joints that have been previously treated with a sealant, such as silicone grease, must be thoroughly cleaned before use. The recommended procedure for removing Dow Corning High Vacuum

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NOTE: The collection efficiencies for PCBs, PCDDs, PCDFs, and PAHs have not been evaluated for this sampling train. However, the train design was based on a validated particulate emissions collection system (EPA Method 5) with the addition of an adsorbent cartridge (packed with XAD-2 resin) to collect vaporous emissions of semivolatile organics. The collection efficiencies of XAD-2 have been evaluated for a large number of compounds including PCBs and PAHs, and XAD-2 was selected for use in the EPA source assessment sampling system (SASS) train.[2,3] XAD-2 was also evaluated for use in a train specially designed for PCB sampling.[4]

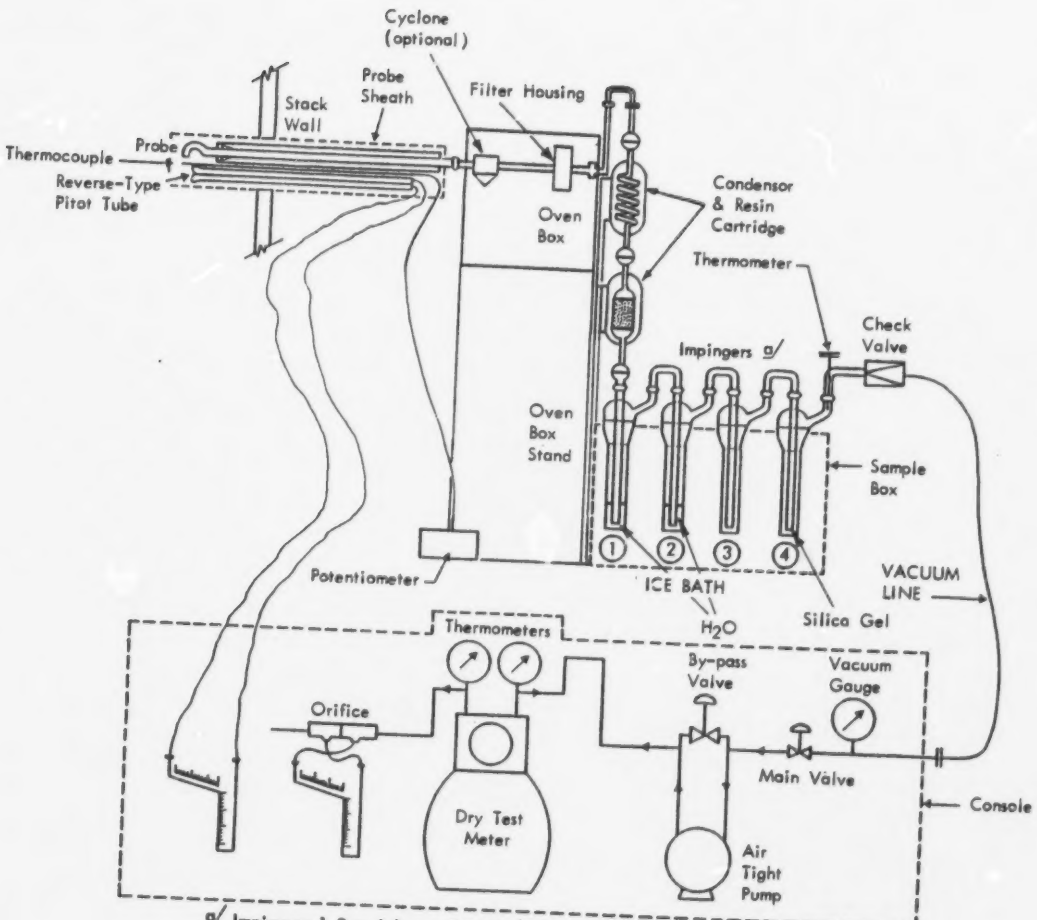


Figure 1. Modified Method 5 train for organics sampling.

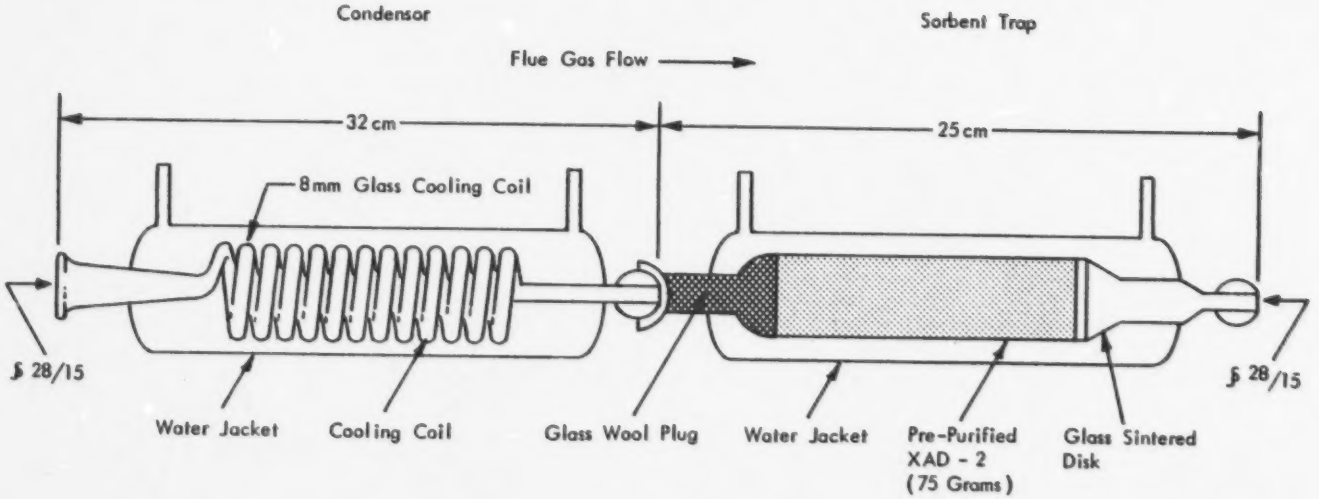


Figure 2. Condensate and sorbent trap modification of the Method 5 sampling train for collection of organic vapors from flue gases.

Silicone Grease involves several steps. Removal of excess grease is accomplished by wiping clean with a rag. The joint is then dipped in warm concentrated KOH for 10 min, rinsed with distilled water, and wiped dry. The last traces of sealant are removed by dipping in chromic acid, followed by rinsing with distilled water and drying. Subsequently, the entire component should be washed in soap (such as Alconox®) and water, followed by at least three liberal rinsings of tap water and then distilled water. All apparatus are then rinsed with acetone (reagent grade) until all traces of water are removed. Finally, the glassware is rinsed sequentially with acetone and cyclohexane (Burdick and Jackson Distilled-in-Glass or equivalent), taking care to contact all sampling surfaces with the solvent. The components should be allowed to dry in a dust-free and organic-free area to minimize contamination of cleaned surfaces. The dried glassware should be protected by covering all open joints and exposed sample-contacting surfaces with solvent-rinsed aluminum foil and by subsequent storage in a closed airtight shipping container until use.

The filter housing gaskets should be cleaned using the same basic procedure with the following modifications. Do not allow non-TFE gaskets to soak in the 15% HNO_3 solution. Following the air drying, place the gaskets in a 225°F oven for 30 min to remove any moisture/solvents. Remove gaskets from the oven and, store in a clean, covered container.

It is highly recommended that TFE filter housing gaskets be used whenever possible as contact with silicon and rubber gaskets can cause significant sample contamination. If non-TFE gaskets must be used, care must be taken to avoid contact with the organic solvents, during sample recovery.

Sample storage containers must also be cleaned prior to use. All sample containers must be amber glass (or wrapped with aluminum foil) with TFE-lined caps. All bottles and sample recovery apparatus must be cleaned with soap and water, water rinsings, acetone rinsing and cyclohexane rinsing as outlined above.

Sorbent resin used in the sampling trains should be precleaned and its cleanliness verified prior to use. The recommended protocol for XAD-2 resins is outlined in the EPA Level 1 Procedures Manual.[3]

All aspects of sampling train assembly should be conducted under the cleanest laboratory conditions possible. To accomplish this, a limited-access field laboratory should be maintained at the site to minimize the possibility of airborne dust problems. Similarly, activities not directly related to train preparation or sample recovery should be done elsewhere. Finally, smoking should not be permitted in the laboratory.

Prior to assembly, all sample-contacting train surfaces should be rinsed with cyclohexane (Burdick and Jackson, Distilled-in-Glass or equivalent). Care must be taken to contact all surfaces with solvent. During assembly it is of vital importance that sealants, such as silicone grease, are not applied to any connecting joints. All train parts must be closely examined for any visual signs of contamination or defects that might induce sample error or downtime problems; corrections will be made if necessary. Leak sealing should

be accomplished using a material that has a high boiling point and high thermal stability, such as the gas chromatography phase, Dow Corning DC 200. Sorbent cartridges must be protected from exposure to light during sampling, sample recovery, and shipping by wrapping each cartridge with aluminum foil.

Pretest Checkout of Sampling Apparatus

Briefly, the checkout involves assembling the entire sampling train as shown in Figure 1 without the probe. The fitting at the inlet of the filter box is sealed and the oven brought to operating temperature. The pump is turned on, and the flow meter gauges are observed for the existence of any appreciable flow. The train must pass the Method 5 standard leak test of less than 0.02 cfm at 15 in. of mercury or 4% of the sampling rate, whichever is less. If an unacceptable leak rate is observed, the operator should (starting at the pump and moving in the direction of the probe) tighten each fitting in order to assure that a loose fitting is not responsible for the leak. If this action does not solve the leak, the system should be leak checked on a modular basis until the problem is pinpointed. Under no condition should a sampling test be conducted with a leak rate in excess of 0.02 cfm at 15 in. Hg.

Flue Gas Sampling Procedures

Standard U.S. EPA methodology for particulate sampling, Method 5, as specified in the Federal Register[1] will be followed.

Two modified Method 5 sampling trains operating simultaneously should be used to traverse points at the center of equal areas within the stack. The number of traverse points and duration of sampling at each point should be provided to the sampling crews. The sampling rates should be adjusted to obtain samples at isokinetic conditions. The sum of flue gas collected each day in the two trains should total 20 m^3 ± 10%.

After the sampling trains are properly assembled and an acceptable pretest leak checkout has been made, preheat the probe and oven to 250°F. The stack temperature, moisture content, and velocity profiles must be determined. Compute the appropriate sampler flow rate and the proper nozzle size using the procedures and calibration curves supplied by the equipment manufacturer.

During the course of the sampling run, scheduled parameter checks should be made on flow rates, temperatures, and pressures. These data should be logged in a sampling record book. Sufficient ice must be kept in the impinger box to chill the condensor and resin trap to keep the impingers cool. At the conclusion of the sampling run, a post leak rate check should be performed.

Sample Recovery

Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. During all rinsing, the approximate volumes of glass-distilled water, acetone, and cyclohexane used should be recorded. This is necessary for the determination of background contributions from the solvents. All organic solvents should be Burdick and Jackson

Distilled-in-Glass or equivalent quality. The wash bottles used for all rinsings should be clean glass or TFE. Other plastic materials are unacceptable due to their potential for sample contamination.

When the probe can be safely handled, wipe off all external particulate matter near the tips of the probe nozzle. Remove the probe from the train and cap off the mating joints of both the probe and the train with solvent-rinsed aluminum foil. Also, cap the outlet of the train assembly after disconnection from the pump. Transfer the probe and train assemblies to the field laboratory for cleanup. This area should be clean and protected to minimize the chance of sample contamination or loss. Inspect the train prior to and during disassembly and note any abnormal conditions. Remove the sorbent trap from the train and cap it off. The cartridge should be transferred to the analytical laboratory intact for further sample recovery.

Rinse the probe with three portions each of water, acetone, and cyclohexane. Brush the entire length of the probe with a natural bristle brush during each rinse. The connecting tube between the sorbent module and the filter housing should then be subjected to sequential rinsings using acetone and cyclohexane, respectively. These rinses should be combined with the probe and filter holder rinses.

The filter particulate is recovered by carefully removing the used filter from the filter housing. Care must be taken to avoid tearing the filter or losing particulate sample. The filter should be stored in a suitable sealed glass container, such that the filter and its contents may be readily removed for weighing in the lab. After removal of the filter, both halves of the filter housing should be subjected to sequential rinsing with acetone and cyclohexane. These rinses should be combined with the preceding rinses. Non-TFE filter housing gaskets should <u>not</u> be rinsed during sample recovery.

When cyclones are employed, the cyclone particulate catch should be recovered and stored in a separate sealed glass container. The cyclone should be rinsed with water, acetone, and then cyclohexane. The rinses should be combined with the other rinses. Similarly, all remaining interconnecting tubing should be rinsed with acetone and cyclohexane. These rinses should be combined with previous rinses.

The contents of the first impinger (aqueous condensate) should be poured into a tared sample bottle. The bottle should be reweighed to ± 1 g and the weight recorded in the sampling record book. The impinger jar should be rinsed with acetone and cyclohexane and the rinses added to the sample bottle. Water accumulated in the remaining impingers should also be determined gravimetrically to ± 1 g.

Upon completion of the train recovery, at least four and possibly five samples should be recovered: (a) the resin cartridge, (b) filter particulate, (c) the first impinger contents, and (d) combined water, acetone, and cyclohexane rinses of the entire train forward of the sorbent trap. A cyclone catch will be the fifth sample if cyclones are employed.

The importance of thorough rinsing cannot be overstressed. Care must be taken to completely contact the interior surfaces of the train with each rinse to quantitatively remove the analyte material. Approximate rinsing volumes of each solvent used for each component should be recorded to allow the accurate determination of solvent background contributions. After all rinsing is completed, the sample bottle should be sealed and the volume of the contents marked to identify possible losses during shipment. All samples should be labeled and logged in the sampling record book as they are recovered. All samples and rinses should be refrigerated at 4°C (or stored in an ice chest) and exposure to light should be minimized during storage and shipment.

SOLID AND LIQUID SAMPLING

All sampling site locations should be clearly and appropriately labeled for easy identification. Also posted at the sample site should be an explanation of any subsample grid scheme to be followed. This serves as a reminder of specific details in subsampling. The crew chief should tour the sampling locations with the sampling personnel prior to the test to verify the collection procedure.

Crew chiefs should provide copies of all sampling schedules for the plant supevisor to post with the plant operations staff. This should provide for any necessary plant staff supervision or assistance in obtaining samples, or in the event of an emergency.

The solid and liquid sampling schedule will start at 0000 hr on the first day of flue gas sampling. Visits should to be made to sample sites as scheduled, and samples taken and placed into prelabeled bottles. Sample and container size required for typical media which should be sampled are given in Table 1. Also included are the recommended number of samples to be collected each day. All samples should be labeled and logged in the sampling record book as they are collected.

Stainless steel trowels, cups, and tongs and glass bottles should be used as necessary in sample capture. Long-handled extensions may be needed to reach some specific areas. Sampling tools should be kept free from contaminants and cleaned with methodology described in this manual. Collection of some samples may require special safety measures such as lab coat, work suit, plant staff or assistance. Safety should be a primary consideration in <u>all</u> sampling operations.

Solid and liquid sampling should continue through all three shifts each day. Sampling staff will have to determine a suitable schedule so all shifts are covered.

COMBUSTION AIR SAMPLING

Combustion air samples should be collected on 75 g of prepurified XAD-2 resin using vacuum pumps equipped with dry gas meters. A sampling rate of 0.75 cfm should be sustained until a total of 10-20 m^3 has been sampled. The resin should be placed in a cartridge similar to that in the sampling train, but without the condensor. The resin cartridge should be wrapped in aluminum

foil to prevent photodegradation of the adsorbed organic compounds. The sorbent cartridge should be capped immediately after sampling has been completed to prevent contamination. This sample should be labeled and stored on ice as soon as possible after collection.

CONTINUOUS MONITORING

Continuous monitoring of the flue gas should be conducted during the period of flue gas sampling to aid in characterizing the efficiency of the combustion process and to provide an indicator of dramatic changes in the unit performance. The parameters monitored should include oxygen, carbon monoxide, carbon dioxide, and total hydrocarbons. The continuous monitoring probe should be inserted into the gas stream inlet to the air pollution control device to mitigate the influence of dilution by ambient air infiltration.

PROCESS DATA COLLECTION

In order to fully characterize the operation of the particular combustion facility it is necessary to collect the engineering data during flue gas sampling. A member of the sampling crew should be assigned to obtain pertinent information concerning the general description and design data and parameters for the power plant and air pollution control equipment that is not sufficiently described from the presite visit. In addition, details and schedules for soot blowing and ash removal during the actual testing period should be recorded. A member of the sampling crew should document any plant breakdown, maintenance, or operating problem during each day's test period that may have an impact on that day's test results. Process engineering data should also be recorded for the megawatt output, steam flow rate, coal loading (rate or total during each test period), and the operation of the electrostatic precipitators during each test period. The electrostatic precipitators should be monitored for operating voltages and amperages, rapping frequency, spark rate, and the number of inoperable units, if any. Information should also be obtained concerning the electrostatic precipitator installed on the unit. Specific parameters include the design volume, temperature, inlet concentration, number of precipitators, field array, gas passages per field, collecting surfaces, collecting surface spacing, face area per precipitator (ft^2), total surface (ft^2), gas velocity, and retention.

QUALITY ASSURANCE PROCEDURES

Calibrations

All sampling equipment should be calibrated prior to testing according to the procedures outlined for Method 5 sampling trains.[1] This should include probe nozzle diameter measurements, pitot tube, and dry gas meter calibrations as well as dial and liquid-filled thermometers and thermocouple-potentiometer system calibrations.

Field Blanks

The collection of field blanks is mandatory to indicate the cleanliness of all sample handling phases. A complete flue gas sampling train should be assembled in the field laboratory for use as a train blank at each plant. The probe and filter oven should be maintained at operating temperatures for a period equivalent to a typical sampling run. At the end of this period the train should be disassembled and the sample components recovered in the same manner as a flue gas sample.

Blanks of each rinsing solvent, as well as unused filters and resin cartridges, must be collected for blank determinations. The resin cartridges and filter blanks should be exposed to the same laboratory environment for the same time intervals as the sample filter and sorbent resin. Similarly, individual samples of acetone and cyclohexane rinse solvents must be collected from the wash bottles for each lot number solvent used. The volume of each solvent blank collected should be approximately equal to the solvent volumes used during the recovery of a sampling train. At least three unused sample bottles of each type should be designated as bottle blanks. All blank materials must be stored in clean sealed glass or TFE containers and treated as samples. Liquid volumes should be marked on the containers to monitor possible shipping losses.

SAMPLE CONTROL AND SHIPPING

An area designated for sample control and shipment preparation should be close to the field laboratory. This area should also have limited personnel traffic. Some stations may require lock and key access if outside of regular plant surveillance. The following shipment preparations should be done by staff on solid/liquid sampling duty.

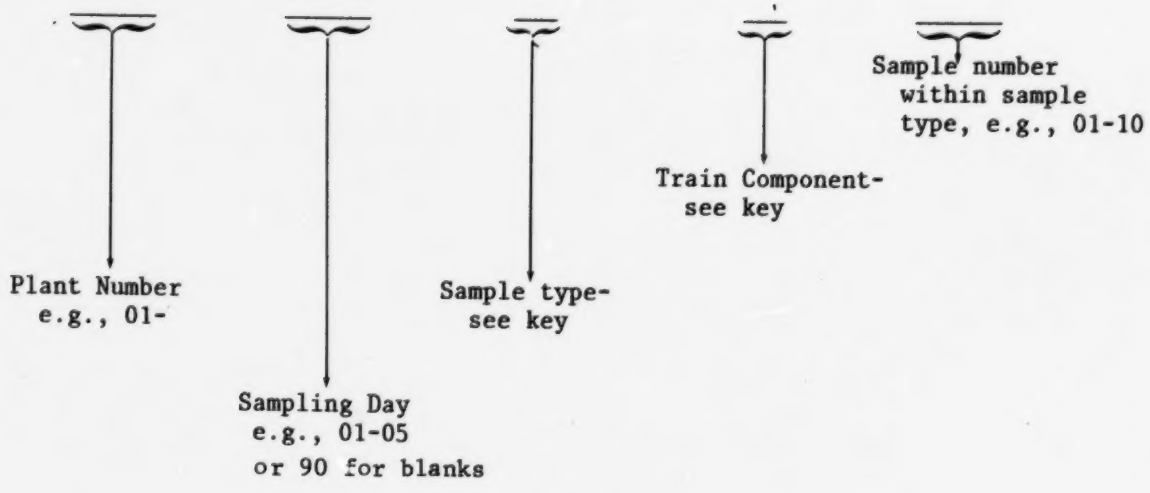
Properly labeled bottles should be supplied to the sample train recovery team. This team should then complete the label and return the full sample bottle and any necessary sampling or recovery remarks to the sample control and shipment operator.

Labels should be provided on computer printout paper and should be grouped by date. Each date should be subgrouped into air sample labels and solid and liquid sample labels. Extra labels should be provided at periodic intervals of the label packet.

All labels should be provided in duplicate. One label should be placed along the left column of a log book page each time a sample is labeled and secured in an ice chest. This will provide chronological entry of sample codes in the log book. Any sampling remarks should be recorded along side the label. Also to be included in this right margin is the sample cooler number in which the corresponding labeled sample has been placed.

MRI's labeling system provides an 8-digit number on each label to be used for primary sample tracking. The label number is defined in Figure 3. The label will also contain a 16-character sample code which provides for easy, accurate identification. This sample code is defined in Figure 4.

LABEL CODE



SAMPLE TYPE

0 - Flue Gas Outlet
1 - Bottom Ash
2 - Control Device Ash (Fly Ash)
3 - Combustion Air
4 - Coal
5 - Refuse-Derived Fuel

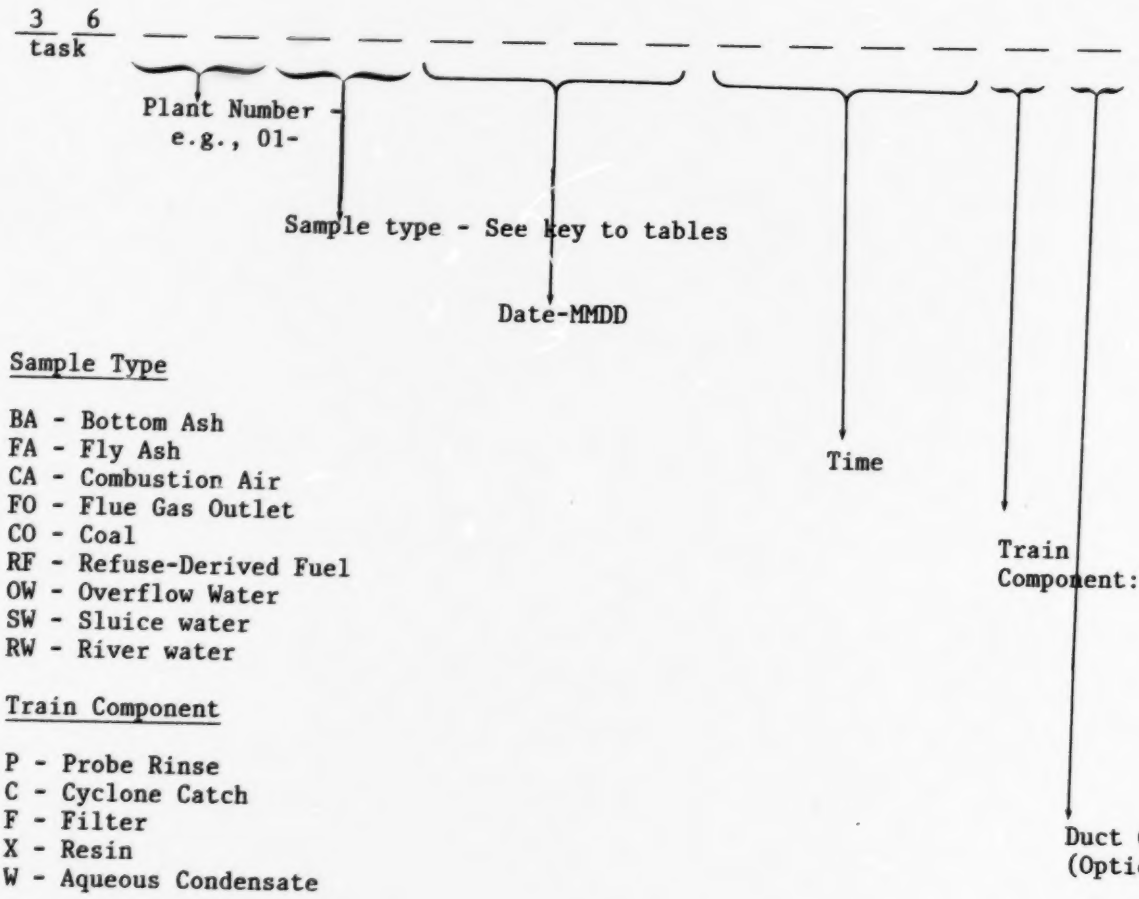
Water sources will be numbered and defined as they are taken.

TRAIN COMPONENT

0 - No Component
1 - Probe Rinse
2 - Cyclone Catch
3 - Filter
4 - Resin
5 - Aqueous Condensate

Figure 3. Eight-digit label code for stationary combustion source samples.

SAMPLE CODES



Sample Type

BA - Bottom Ash
FA - Fly Ash
CA - Combustion Air
FO - Flue Gas Outlet
CO - Coal
RF - Refuse-Derived Fuel
OW - Overflow Water
SW - Sluice water
RW - River water

Train Component

P - Probe Rinse
C - Cyclone Catch
F - Filter
X - Resin
W - Aqueous Condensate

Figure 4. Sixteen character sample code for stationary combustion source samples.

All labels should be prepared so that minimum amount of additional information must be entered in the field. The time of sample capture, any subsampling location designations, and the sampler's name are typically the only entries. All entries must be made on the duplicate label as well.

Labels should be self-adhesive. In addition, 1-1/2 to 2 in. transparent tape should be overlaid on the completed label placed on sample containers as a precautionary measure.

All liquid samples should have the volume marked on the side of the shipping container with a permanent marker. This should allow losses of sample from handling and shipping to be noted.

Samples other than those in amber glass bottles will require special packaging. These are given in the following two paragraphs.

Modified Method 5 Particulate

The particulate filters should be returned to their original containers (petri dishes) when sampling is completed. Each petri dish should be taped shut using masking tape. The identification label should be placed on the top center of each dish. The filter and dish number should be included on both labels. The dish should be wrapped in aluminum foil and sample ID number (from label) copied onto top side. The petri dish should be carefully sealed in a ziplock bag, with a minimum air space. Care must be taken to ensure that filters are returned to the original containers since it is necessary to know the predetermined weight of each specific filter.

Resin Cartridge

The sample identification label should be affixed to the aluminum foil covering. The entire cartridge should be wrapped in an adequate amount of bubble pack (bubbles to inside) with cartridge ends capped securely with glass balls. A lab marker should be used to copy the label ID number on to the outside of the wrap.

Sample Custody Documentation

A chain-of-custody record should be prepared for every sample. The custody sheet should be initiated in duplicate immediately after the sample has been labeled. It should include the sample label number and the sampler's signature. At the time of sample shipment, the record should be signed and the time and date should be noted. The original copy of the chain-of-custody record should be enclosed in the sample container. The yellow copy should be retained by the crew chief until the samples are received and logged in at the analytical laboratory.

When the container arrives at the laboratory, the person who will be preparing the samples should receive (take custody of) it. That person should then open the shipping container and check each sample for damage or tampering. This person should then sign all the enclosed chain-of-custody records and note any damage or indication of tampering.

Whenever custody of the sample is transferred from one person to another, the person relinquishing custody of the sample should sign the chain-of-custody form and note the time and date. The person receiving the sample should do the same. The person having custody of the sample should have sole control of access to the sample.

Figure 5 is an example of a chain-of-custody record used by MRI.

4900-A36

Midwest Research Institute
425 Volker Boulevard
Kansas City, Missouri 64110

MRI

CHAIN OF CUSTODY RECORD

Label No.:	Samplers: (Signature)

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time	Comments (Changes, Volume Removed, Dilutions, etc.)

Figure 5. Sample custody transfer sheet used by MRI.

SECTION 3

SAMPLE ANALYSIS

The analytical procedures described in this section were developed during a pilot study of stationary combustion source facilities. The primary objective of this section is to ensure that the extraction and analysis of samples from other such facilities is coordinated and consistent for all laboratories involved in possible future Exposure Evaluation Division programs.

Each of the different types of samples should be combined into daily composites, extracted, and analyzed by capillary gas chromatography using flame ionization and Hall (halide mode) electrolytic conductivity detectors (HRGC/Hall-FID) and by capillary gas chromatography/mass spectrometry (HRGC/MS) using fused silica columns. The analytical scheme presented in Figure 6 should be followed to determine the presence of various compounds in sample extracts. Both qualitative and quantitative results are expected for the range of polycyclic and chlorinated organic compounds determined by these procedures. A rigorous quality assurance/quality control program has been outlined and should be considered by other laboratories participating in similar analytical efforts. In addition, a procedure to determine total organic chlorine (TOCl) is described. This technique may be beneficial for pilot studies to provide a more sensitive means of identifying the presence of chlorinated polycyclic compounds.

GENERAL ANALYTICAL PROCEDURES

All solvents should be Burdick and Jackson, Distilled-in Glass, pesticide grade quality, or equivalent. Glass wool, boiling chips and anhydrous sodium sulfate should be pre-extracted with a hexane-acetone mixture or benzene. The anhydrous sodium sulfate should be extracted with the hexane-acetone azeotropic mixture, air dried, heated at 110°C for several hours, and finally baked at 650°C for at least 2 hr. It is important to allow the extracted Na_2SO_4 to dry thoroughly before baking at high temperature to avoid explosions in the high temperature oven.

All glassware that will be used in handling the samples and extracts should be cleaned first with soap and hot water, rinsed thoroughly with hot water, followed by distilled water. Acetone (reagent grade) should be used to rinse glassware for removal of all traces of water and final rinses with acetone and cyclohexane should be required. If blanks are a problem, the glassware should be baked at 400°C for at least 8 hr prior to use.

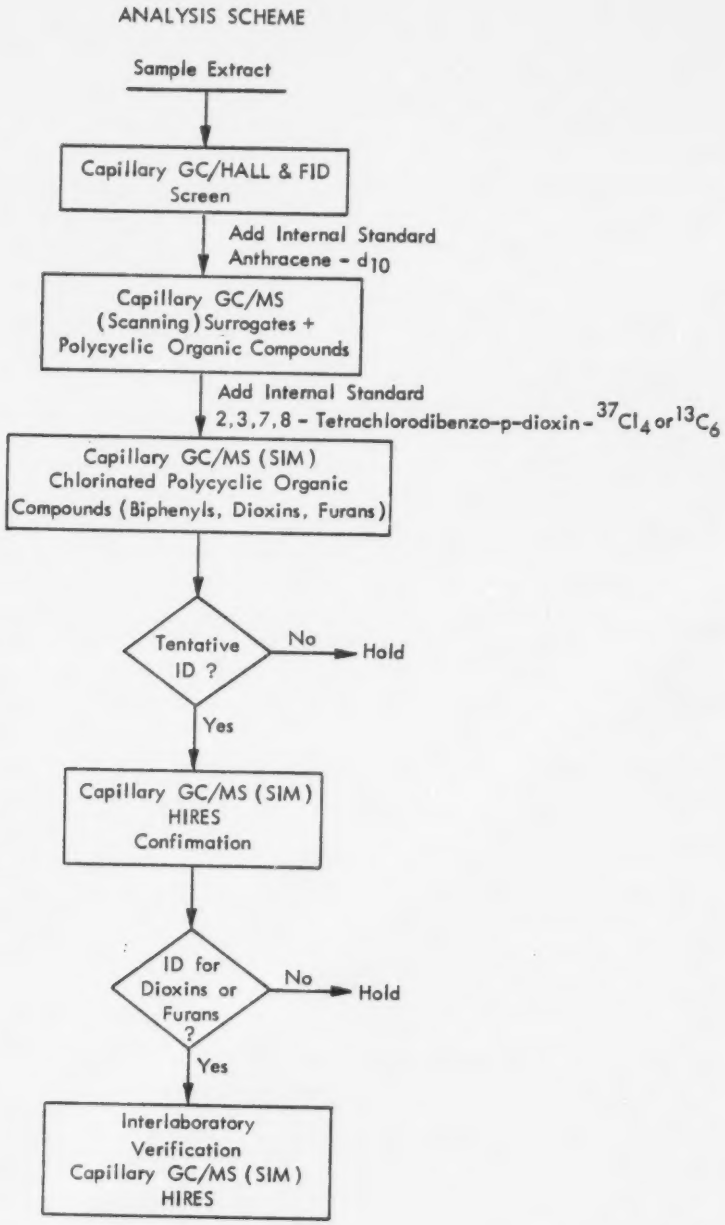


Figure 6. Analysis scheme for sample extracts.

EXTRACTION

The sample extraction methods described below have been developed to maximize recovery of a wide range of PAH compounds and polychlorinated aromatics. Each sample should be spiked with labeled surrogate compounds prior to extraction for component recovery determinations.

Solid Samples

All solid samples should be Soxhlet extracted for 8 to 24 hr using benzene (Burdick and Jackson, Distilled-in-Glass or equivalent) as the solvent. Solid samples include XAD-2 resin, filters from the filter catch, and the cyclone catch from the modified Method 5 train, control device ash (fly ash), bottom ash, and fuel.

Samples from the Modified Method 5 sampling train should be extracted simultaneously. For example, filters from the filter catch taken for a single day's operation may be combined and extracted in the same Soxhlet apparatus. However, sampling train materials should not be mixed (e.g., XAD-2 resin, filters, cyclone material) since XAD-2 resins may be reclaimed. The filters from the filter catch should be weighed prior to extraction. The weight of the collected particulate matter should be calculated from measurements for each filter made prior to sampling the field. No attempt should be made to achieve constant weights for the filter samples. Also, the particulate material obtained from the cyclone and probe rinses should be weighed prior to extraction.

Control device ash and bottom ash should be individually composited for each sampling day and 20 g each of these media Soxhlet extracted with benzene for 8 to 24 hr. Prior to extraction, 10 ml of organic-free water should be added to the control device ash. If the bottom ash is dry, 10 ml of water should also be added to wet the material before beginning the extraction. An inert material, such as Chromosorb W can be added to ash samples to promote more efficient solvent flow through the sample.

Coal (10 g) should be Soxhlet extracted with benzene (8 to 16 hr). Large mesh coal samples should first be ground to a powder using a ceramic mill with stainless steel balls. Refuse-derived fuel will be evaluated when homogeneous samples are available. This material (10 to 20 g) should also be milled and Soxhlet extracted with benzene as the solvent for periods of 8 to 24 hr. This extract should be washed three to four times with 100-ml aliquots of organic free water.

All extracts should be dried by passage through short columns of anhydrous sodium sulfate. The dry extracts should be concentrated in Kuderna-Danish appartus to approximately 5 ml. The extracts from the various components of each day's flue gas sample should be combined and reduced to 5 ml in a Kuderna-Danish evaporator. The extracts should be further concentrated to 1 ml with a gentle stream of purified nitrogen. If solids precipitate from extracts during concentration, slowly dilute and stir the extract to redissolve the solids. Do not attempt to further concentrate the extract.

Aqueous Samples

Aqueous samples may be obtained as plant influents and effluents, as well as from the combined train rinses from flue gas sampling and the aqueous condensate from the first impinger. The combined train rinses should include water, acetone, and cyclohexane. This sample should be shaken vigorously and the organic layer removed using a separatory funnel. Two 60-ml aliquots of cyclohexane should be used to extract the aqueous solution. The cyclohexane extracts should be combined, dried by passing through a short column of anhydrous sodium sulfate, and finally combined and reduced with other extracts from the modified Method 5 sampling train. If emulsions are formed during the extraction, 2 to 3 g of sodium sulfate can be added to the mixture to promote adequate separation. The extracts should be dried and concentrated in the same manner as extracts from solids.

EXTRACT FRACTIONATION/CLEANUP

Sample extracts, particularly the flue gas samples, may contain considerable interferences that may present problems in the effective analysis of such compounds as polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans or the surrogate compounds. These extracts should be cleaned by EPA Method 613 to simplify the sample matrix prior to analysis for polychlorinated dibenzo-p-dioxins and dibenzofurans. Since other polycyclic organic compounds may be lost by this cleanup procedure, and it may be necessary to fractionate a portion of sample by other techniques, such as silica or florisil adsorption chromatography, prior to GC/MS analysis. Only half of a sample extract should be submitted to any fractionation scheme. It is also important to measure the exact volume of a sample extract subjected to cleanup to ensure valid quantitation of specific compounds on the final aliquot.

EXTRACT ANALYSIS

HRGC/Hall-FID

Each extract should be screened first for the presence of halogenated organic compounds by capillary gas chromatography separation with a Hall electrolytic conductivity detector operated in the halogen specific mode. Fused silica capillary columns, 30 m in length and coated with SE-54 [1% vinyl in poly(methylphenylsiloxane) previously deactivated by silylation], 0.25 mm ID, will be used for gas chromatography separations of all extracts.

On-column and direct injection techniques are preferred, but Grob-type splitless injectors may be used, if necessary. The extracts should be chromatographed using the following temperature program: isothermal at 60°C for 2 min, increase temperature at 10°/min to 300°C and hold isothermally for 15 min. The qualitative results from these analysis should be useful in identifying the presence of halogenated compounds by gas chromatography/mass spectrometry analyses. Large halogen responses at specific retention times should identify the regions of the chromatograms where interpretation of the mass spectra data may lead to the identification of halogenated compounds.

HRGC/MS (Scanning)

The correlation of the mass spectral data with halogen responses from the Hall electrolytic conductivity detector is necessary for positive identification of chlorinated organic compounds in the sample extract. Therefore, it is necessary to duplicate gas chromatography conditions for the two methods. Fused silica capillary columns coated with SE-54, 15 to 30 m in length, should be used for all scanning HRGC/MS studies. On column, direct, or Grob-type splitless injections should be used and the same temperature program used for HRGC/Hall-FID should be followed.

Mass spectra should be acquired over the range of m/e 40 to 500 at a rate of 1 to 1.2 sec/scan. The spectral data from these analyses should be used for both qualitative and quantitative determinations. The compounds that are positively identified and are of sufficient concentration should be quantitated by peak area from the total ion chromatogram versus the peak area of the appropriate internal standard.

HRGC/MS Selected Ion Monitoring (HRGC/MS-SIM)

The selected ion monitoring (SIM) technique should be used to determine the presence of chlorinated dibenzodioxins or dibenzofurans. Preliminary indications of the presence of these compounds may be evident from results of the HRGC/Hall-FID and HRGC/MS (scanning) experiments. However, the HRGC/MS-SIM technique has greater sensitivity for determination of these compounds in the sample extracts. Selected ions characteristic of the mono- through octachloro PCDDs, PCDFs, and PCBs should be monitored by this technique. The criteria for the identification of these analytes in any extract will be dependent on the coincidence of peaks in the extracted ion current plots of the characteristic ions at the appropriate retention times and on the characteristic relative intensity ratios of these selected ions. Table 2 lists the HRGC/MS-SIM ions that should be used to identify the presence of the dibenzodioxins, dibenzofurans, and biphenyls. Fused silica capillary columns coated with Carbowax 20M or other polar materials may be used for isomer specific SIM analyses for polychlorinated dibenzodioxins and dibenzofurans.

HRGC/MS High Resolution Mass Spectrometry (HRGC/HRMS)

The tentative identification of dibenzodioxins and dibenzofurans by HRGC/MS-SIM in any extract should be confirmed by HRGC/HRMS. This procedure should be used to verify the presence of these compounds in any sample extract. Positive identifications of chlorinated dibenzodioxins or chlorinated dibenzofurans by HRGC/HRMS should be supplemented by verification by other accredited laboratories with HRGC/HRMS capabilities. All HRGC/HRMS studies should employ fused silica capillary columns coated with either SE-54, Carbowax 20M, or other materials capable of providing equivalent or better chromatographic resolution.

TABLE 2. GC/MS-SIM IONS

Number of chlorines	Biphenyls	Dibenzofurans	Dibenzo-p-dioxins
1	188/190	202/204	218/220
2	222/224	242/244	252/254
3	256/258	270/272	286/288
4	290/292	304/306	320/322
5	324/326	338/340	354/356
6	360/362	374/376	390/392
7	394/396	408/410	424/426
8	428/430	442/444	458/460
9	462/464	-	-
10	498/500	-	-

Total Organic Chlorine (TOCl) Measurements

TOCl measurements may be used in a primary sensitive screen of the chromatographable organically bound halide contents of extracts prepared for GC/MS analysis in tiered analytical schemes. The TOCl procedure is a simplified gas chromatographic method using a Hall electrolytic conductivity detector in the halide mode. A short packed GC column (typically 1-2 in. x 1/4 in. ID) and a rapid temperature program are used to elute all chromatographable compounds with volatiles equal to or greater than dichlorobenzenes as a single peak. The area of this peak constitutes the TOCl response which is quantitated as chloride against a mixture of chlorinated compounds (typically a PCB mixture such as Aroclor 1254). The typical method senstivity is 0.25-2 ng chloride.

QUALITY ASSURANCE (QA) PROCEDURES

The positive identification and quantitation of specific compounds in this assessment of stationary conventional combustion sources is highly dependent on the integrity of the samples received and the precision and accuracy of all analytical procedures employed. The QA procedures described in this section were designed to monitor the performance of the analytical methods and to provide information to take corrective actions if problems are observed. These procedures are summarized in Table 3.

Field Blanks

The field blanks should be submitted as part of the samples collected at each particular testing site. These blanks should consist of materials that are used for sample collection and storage and are expected to be handled with exactly the same procedure as each sample medium.

Method Blanks

A method blank should be prepared for each set of analytical operations. This will evaluate contaminations and artifacts that are derived from glassware, reagents and sample handling in the laboratory. Method blanks should be evaluated by each laboratory for solid and aqueous sample extractions.

Recovery Spikes

Surrogate compounds should be added to all samples prior to extraction to provide an accurate record of analyte recovery. Specific analytes should be used for method development procedures. In either case, duplicate samples should be prepared. The surrogate compounds should include napthalene-d_8 and chrysene-d_{12}. The other compounds that will be used as surrogates include pentachlorophenol-$^{13}C_6$, 1,2,4,5-tetrachlorobenzene-$^{13}C_6$, and 3,4,3',4'-tetrachlorobiphenyl-d_6.

Internal Standards

Each concentrated extract should be spiked with anthracene-d_{10} prior to analysis by HRGC/MS in the scanning mode. This should allow for adequate quantitation of specific analytes in the extracts once proper response ratios have been established using standard solutions. This internal standard should be added to the extracts to yield a concentration in the range of the analytes and the surrogate compounds. This internal standard can also be used to determine relative retention times in any particular chromatograms, and this provides another means of analyte identification. Stable isotope labeled isomers of tetrachlorodibenzo-p-dioxin and tetrachlorodibenzofuran should be used for selected ion monitoring methods for these specific compounds. The isotope label should provide sufficient distinction of the internal standard and the actual isomers present in the sample extracts.

Reference Materials

A reference ash will be prepared by compositing ash from several facilities by MRI. Portions of this ash may be sent to interested laboratories as a means of evaluating interlaboratory performance. Two samples of spiked reference ash and two samples of unspiked reference ash will be submitted to all laboratories. These samples will be extracted and analyzed with the same procedure used for all other samples.

Capillary Column Performance Tests

The optimum performance of the fused silica capillary columns coated with SE-54 is an integral function of the separation and identification of specific compounds in the sample extracts. Therefore, each laboratory should frequently evaluate the performance of capillary columns used for extract analysis. Grob-type test mixtures should be used to evaluate each column used for GC/Hall and GC/MS studies. A test mixture prepared with halogenated compounds should be used to test capillary columns with Hall electrolytic conductivity detectors.

TABLE 3. SAMPLING AND ANALYSIS QUALITY ASSURANCE

Field blanks	
Method blanks	
Recovery spikes -	analytes
	surrogates
	d_8-napthalene
	d_{12}-chrysene
	$^{13}C_6$-1,2,4,5-tetrachlorobenzene
	d_6-3,4,3',4'-tetrachlorobiphenyl
	$^{13}C_6$-pentachlorophenol
Internal standards -	d_{10}-anthracene
	^{37}Cl-TCDD or ^{13}C
	^{37}Cl-TCDF
Reference materials	
Capillary column performance checks	
Interlaboratory verification	

The parameters that should be monitored include separation number (Tz) for a homologous series of compounds, the height equivalent for theoretical plates (HETP), the number of theoretical plates (N), peak asymmetry, adsorption ratios, and pH of the column. Peak asymmetry is calculated for each peak of the test mixture from the formula:

$$AS = \frac{W_b}{W_f} \times 100$$

Where W_b and W_f are the back and front baseline widths of the peak measured from a line bisecting the peak maximum. Adsorption ratios are determined by comparison of the peak height for a compound susceptible to adsorption with that of an inert compound. The pH of a column can be determined from the ratio of the peak heights of equivalent quantities of an acid and a base in the test mixtures.

The capillary columns should be evaluated immediately upon installation and at least once per week. The capillary columns should be rejected for poor performance as related to separation number, adsorptivity, and pH. The columns should be tested more frequently if drastic deterioration of the column is noted in a 1-week time span.

Interlaboratory Verification

All extracts in which polychlorinated dibenzodioxins and dibenzofurans are identified by HRGC/MS or HRGC/HRMS should be submitted to other laboratories for confirmation of these identifications by HRGC/HRMS.

SECTION 4

DATA REPORTING

The following section provides examples of the pertinent data that should be reported for characterization of polycyclic organic matter from stationary combustion sources. Examples are given for methods of sample tracking through the entire organic compound analyses, reporting of analytical data for surrogate compounds and the inputs and emissions of particular analytes, and the reporting of the engineering process data necessary for describing flue gas sampling methods and the actual combustion process. It is highly recommended that all laboratories involved in combustion facility characterizations for polycyclic organic materials adhere as closely to these reporting guidelines to facilitate comparision of data from several sources.

SAMPLE TRACKING (ANALYTICAL)

Sample tracking sheets should be used by all analytical laboratories to monitor the status of sample analyses. An example of the tracking sheet is shown in Table 4. The sample numbers illustrated are truncated when samples for 1 day's operation are composited. The pertinent information presented in this sample number includes the task number (36), plant number, and the date sampled.

The sample tracking sheets should be initiated upon receipt of the samples from the field stations. The information on the sheets should include dates samples were received and dates the samples were composited and extracted. The other designations should indicate the extent of analysis for each composited sample, i.e., screening sample by HRGC/Hall-FID, HRGC/MS, and HRGC/MS-SIM. Other remarks can be added as required. For example, PAH, PCDD, or PCDF might be added to indicate that polynuclear aromatic hydrocarbons, chlorinated dibenzodioxins, or chlorinated dibenzofurans have been tentatively identified in particular extracts. Likewise, other abbreviations might be added for identification of chlorinated benzenes or phenols. Target may be used to indicate that analysis for specific compounds has been completed. Major will be used to specify that the major components of a sample have been investigated.

DATA MANAGEMENT AND REPORTING

The data generated for the target compounds and the major components of each sample extract should be presented to MRI in two forms. Assessment of the QA program should be accomplished by reporting percent recovery of the surrogate compounds in a specific extract together with the concentrations of the target and major compounds in the extracts. Concentrations for the target

TABLE 4. SAMPLE TRACKING SHEET

Sample no.	Received	Compos-sited	Ex-tracted	HRGC/ Hall-FID	HRGC/MS (Scanning)	HRGC/ MS-SIM	To MRI HRGC/ HRMS
36/03/FA/0330	0415	0418	0418	4-20	4-23 Target	4-27 Target	4-29
36/03/BA/0330	0415	0418	0418	4-20	4-23 Major Target	4-27	
36/03/CA/0330	0415	0418	0418	4-20	4-25	4-27	
36/03/FO/0330	0415	0419	0419	4-21	4-25		
36/03/CW/0330	0415	0419	0419	4-21	4-25		

compounds should be reported for all extracts. If a compound is present but cannot be quantitated, it should be reported as less than the detection limit. If a compound is not detected, it should be reported as not detected. The data for QA will be reported as shown in Table 5. The percent recovery should be reported for the surrogate compounds only.

TABLE 5. ANALYTICAL DATA REPORTING SHEET

Day 1 Bottom ash - Composite ID No.

Surrogate compounds	Concentration	% Recovery
d_8-Naphthalene		
c_{12}-Chrysene		
$^{13}C_6$-1,2,4,5-Tetrachlorobenzene		
$^{13}C_6$-3,4,3',4'-Tetrachlorobiphenyl		
$^{13}C_6$-Pentachlorophenol		
Analyte Compounds		
1.		
2.		
3.		
4.		

The other method for reporting the data should follow the concentration of a particular compound in all sample matrices. Sample concentrations should be grouped according to inputs and outputs of the plant. This data report should be presented as shown in Table 6, representative for particular compounds over five composited sampling days.

The data supplied by the engineering report will be used to determine the mass flow inputs and emissions for the various sample media. The summary of the flue gas sampling and the continuous monitoring data should be tabulated as shown in Table 7.

TABLE 6. TOTAL INPUTS AND EMISSIONS

Composite day	Inputs: Combustion: Mass input (dscm/hr)	Inputs: Combustion: Conc. (ng/g)	Inputs: Combustion: CA input (mg/hr)	Inputs: Coal: Feed rate (kg/hr)	Inputs: Coal: Conc. (ng/g)	Inputs: Coal: Coal input	Emissions: Flue gas: Mass emissions (dscm/hr)	Emissions: Flue gas: Conc. (ng/dscm)	Emissions: Flue gas: FO emissions (mg/hr)	Emissions: Bottom ash: Mass flow (kg/hr)	Emissions: Bottom ash: Conc. (ng/g)	Emissions: Bottom ash: BA emissions (mg/hr)
I												
II												
III												
IV												
V												
Mean $\bar{x}$												
Standard deviation												

Composite day	Emissions: Fly ash: Mass flow (kg/hr)	Emissions: Fly ash: Conc. (ng/g)	Emissions: Fly ash: FA emissions (mg/hr)	Miscellaneous outputs (water): Mass flow (ℓ/hr)	Miscellaneous outputs (water): Conc. (ng/ℓ)	Miscellaneous outputs (water): WO emissions (mg/hr)	Total inputs (mg/hr)	Total outputs (mg/hr)
I								
II								
III								
IV								
V								
Mean $\bar{x}$								
Standard deviation								

TABLE 7. DAILY FLUE GAS SAMPLING DATA

Date (1980)	Test no.	Train no.	Sample Volume DSCF	Sample Volume DSCM	Gas composition[a] O_2 %	CO_2 %	CO ppm	THC ppm	Stack temperature °F	Molecular weight	Moisture %	Velocity ft/sec	Gas flow ACFM	Gas flow DSCFM	Gas flow DSCMM	Isokinetic rate %
	1															
	2															
	3															
	4															
	5															

a Average during test period.

SECTION 5

REFERENCES

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4. Haile, C. L., and E. Baladi, "Methods for Determining the Total Polychlorinated Biphenyl Emissions from Incinerator and Capicator- and Transformer-Filling Plants," NTIS No. PB-276 745/761 (1977).